

The Effect of Cold-Drawing on the Creep Behavior of a Nickel-4.2 Percent Aluminum Alloy

William D. Jenkins and William A. Willard

Institute for Materials Research, National Bureau of Standards, Washington, D.C.

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Creep tests were made at 700, 900, and 1200 °F (644, 755, and 921 °K) on a nickel-4.2 percent aluminum alloy (Duranickel) initially as cold-drawn and as cold-drawn and age-hardened. Microstructural changes occurring during creep are correlated with the flow, fracture, and ductility of the specimens. Light and electron microscope observations indicate that increases in creep strength were associated with the formation of rather straight discontinuous slip bands, a well defined subgrain structure, small precipitates of Ni₃Al, and a general distribution of these precipitates throughout the grains. Creep strength at 1200 °F was relatively unaffected by prior cold-drawing or aging. Inter-crystalline cracking, accompanied by low ductility, was evident at 900 and 1200 °F and slow creep rates; whereas transcrystalline cracking and high ductility values were observed at 700 °F.

Key Words: Cold-drawing, creep, nickel alloys, Duranickel, nickel-aluminum precipitates, high temperatures, deformation.

1. Introduction

A comprehensive investigation designed to evaluate the influence of stress, strain, strain rate, temperature, and structure on the rheological properties of a precipitation-hardening nickel-4.2 percent aluminum alloy (Duranickel*) has been in progress for the last few years at the National Bureau of Standards. The tensile and creep properties of annealed and annealed and aged Duranickel have been previously published [1, 2].¹ The present phase of the investigation describes (1) the influence of cold-drawing on hardness and creep behavior; (2) the effect of prior aging on creep characteristics; (3) the influence of creep on aging; (4) the role of creep rate and microstructure on crack formation and fracture. In addition, data, previously published [2] describing the temperature dependence of creep behavior of annealed and annealed and aged Duranickel are included for comparison purposes.

annealed at 1725 °F (1213 °K), then cold-drawn by the manufacturer to give a tensile strength of 127,000 psi and a hardness of 27 Rockwell "C". Specimens, 1/4 in. in length, were cut from the bar and aged in vacuo for 16 hr at temperatures ranging from 700 to 1250 °F (644 to 948 °K). The specimens age-hardened above 900 °F (755 °K) were cooled at a rate of 25 °F/hr to 900 °F, then furnace cooled to room temperature.

TABLE 1. Chemical composition (percentage by weight) of the Duranickel used

Nickel.....	93.94
Aluminum.....	4.20
Silicon.....	0.66
Titanium.....	.39
Copper.....	.31
Manganese.....	.28
Iron.....	.19
Carbon.....	.15
Sulfur.....	.003

Determinations made at the National Bureau of Standards.

2. Material and Testing Procedure

The chemical composition of the metal used in this investigation is given in table 1. All the bars were processed from the same heat as that previously used [1, 2] and were supplied by the Huntington Alloy Products Division of the International Nickel Company in the form of 7/8-in. round rods, approximately 15-ft long. The bars were hot-rolled, descaled, and

Specimens age-hardened at 900 °F and below were also furnace cooled. Room-temperature hardness surveys indicated maximum hardness at 1100 °F (866 °K) was attained with a 16 hr aging time (fig. 1C). Above this temperature at this time the metal appeared to be overaged (fig. 1A). In order to ascertain the effect or rate of cooling on the initial structure and hardness of the cold-drawn metal, specimens were heated in air for 16 hr at temperatures ranging from 700 to 1200 °F (644 to 921 °K) and water quenched (fig. 1A). Room temperature hardness surveys indicated the hardness values for each temperature were lower than those obtained by furnace cooling. Another

*Trade name International Nickel Company.

¹ Figures in brackets indicate the literature references at the end of this paper.

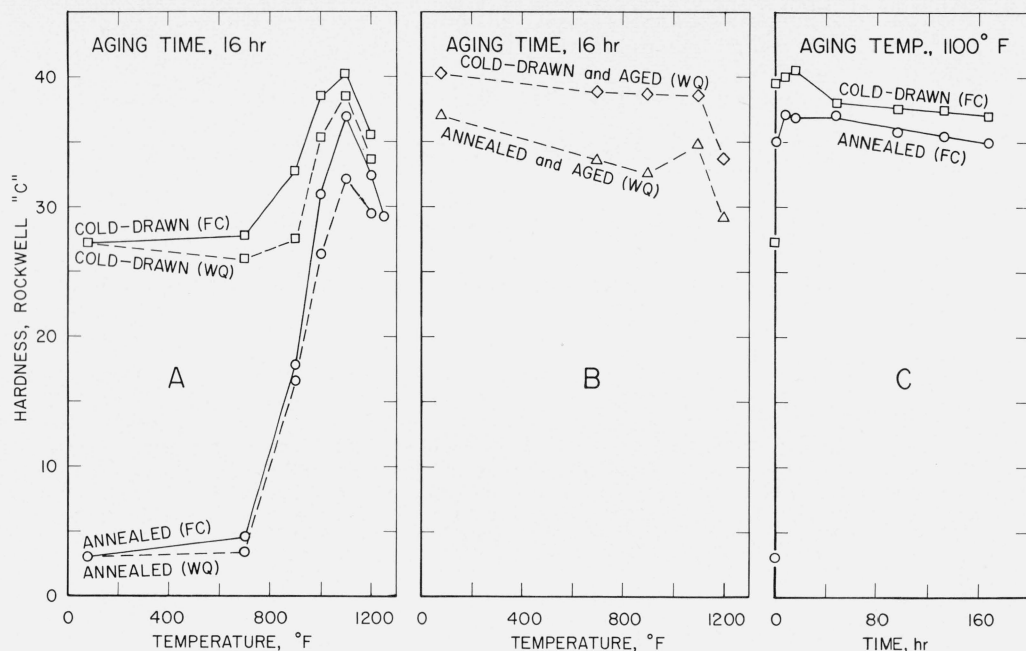


FIGURE 1. Effect of aging temperature and time and rate of cooling on the hardness of Duranickel.
FC—furnace cooled; WQ—water quenched.

group of cold-drawn specimens was aged at 1100 °F for 16 hr and furnace cooled. Then they were heated in air to temperatures of 700, 900, 1100, and 1200 °F and held for 16 hr before water quenching. This procedure was used in order to obtain more stable microstructures in the specimens. Room-temperature hardness values indicated a softening effect for the cold-drawn and aged specimens (fig. 1B). The same general trend was also observed for the annealed and aged specimens (fig. 1B).

Cold-drawn rods were cut into specimens 6¼-in. in length for creep testing. These were divided into two groups: one group to be tested in the cold-drawn condition, the other group to be tested in the aged condition (aged at 1100 °F for 16 hr and furnace cooled). Room-temperature tensile properties were determined by the authors for the annealed, cold-drawn and aged specimens (table 2).

The creep specimens had a reduced section of 0.252-in. diam over a 2-in. gage length and the ends were

machined with ¾ in. × 10 threads. The reduced section was finished by grinding and then polished in the axial direction, using No. 400 Alumina polishing paper. Before testing, each creep specimen, cold-drawn or cold-drawn and aged, was heated in a tube furnace to the desired temperature and equilibrated at temperature for 16 hr. It was then tested at temperature under constant load. The creep-rupture machines used were of a multiple lever type with motorized jacks to prevent shock loading. They were recently calibrated at room temperature and the errors for the stresses used were less than 0.5 percent. The temperature of each of the specimens was controlled to an accuracy of ± 2 °F. Strain-time data were obtained from electric contact follow-up type extensometers attached to shoulders of the specimens. The sensitivity of extension measurement was approximately 0.00005 in.

3. Metallography

Most of the specimens tested to fracture in creep were subjected to a metallographic examination. In general, it was observed that transcrystalline cracks having a round or elliptical shape were predominant at 700 °F. A mixture of transcrystalline and intercrystalline cracks was evident at 900 °F. The intercrystalline cracks were more populous at the slower creep rates. At 1200 °F, the cracks were almost exclusively intercrystalline.

TABLE 2. Room temperature tensile properties of Nickel-4.2 percent aluminum alloy

Condition	Upper yield stress	Lower yield stress	Yield strength (0.2% offset)	Tensile strength	Elongation	Reduction area
	psi	psi	psi	psi	Percent	Percent
Annealed.....	45,900	45,700	104,200	47	77
Annealed and aged.....	122,200	183,000	20	48
Cold-drawn.....	101,400	126,600	22	65
Cold-drawn and aged.....	145,500	188,000	18	52

Four specimens, two each of the annealed and aged and the cold-drawn and aged metal, were selected for a comprehensive metallographic examination after being tested to fracture in creep. The microstructures are shown in figures 2 to 7. Microstructures of the alloy in the annealed and annealed and aged conditions were previously shown [1].

The basis for selection was (1) these specimens illustrate the effect of cold-drawing on the aged micro-

structure; (2) they represent the microstructures associated with the extremes of temperatures used (700 and 1200 °F); (3) they show the effects of slow creep rates on changes in microstructure; and (4) they represent microstructures that differed significantly from those previously shown for fast creep rates [1, 2]. Cracks, formed at 700 °F, are illustrated by the photomicrographs in figure 2A and B. Cracks formed at 1200 °F are shown in figure 2C and D. More cracks

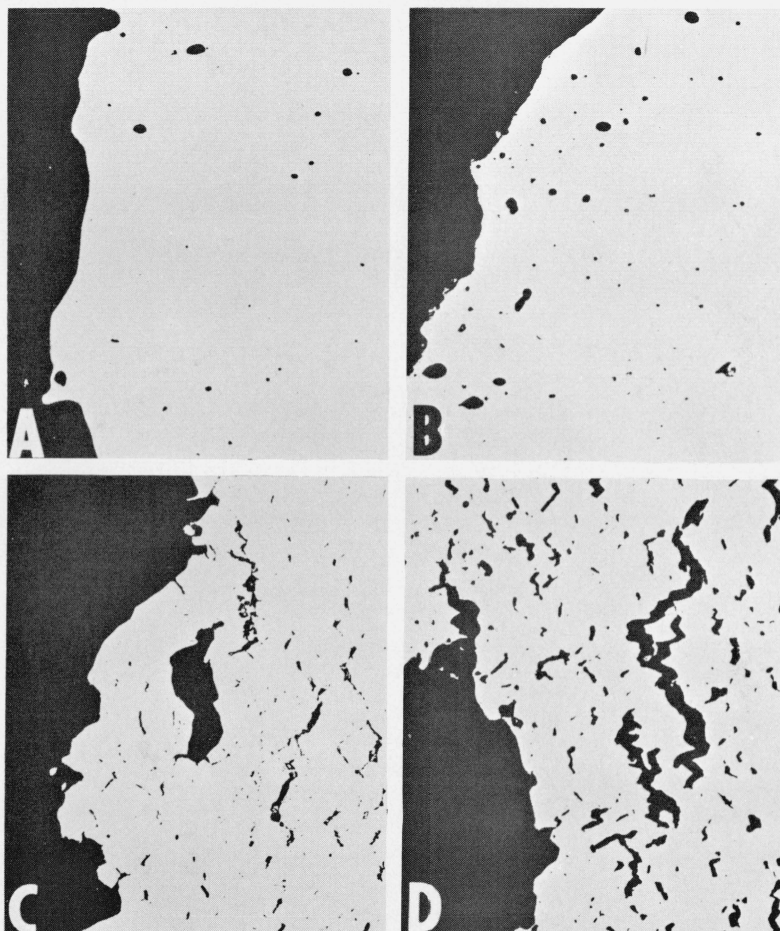


FIGURE 2. *Relation of test conditions to number and distribution of cracks in fractured creep specimens.*

Longitudinal sections, at axis, near fracture, unetched. X 83.

Condition	Test temperature	Fracture time	Reduction of area
	°F	hr	%
A Annealed and aged	700	1367.2	5
B Cold-drawn and aged	700	1068	13.5
C Annealed and aged	1200	2059.7	6
D Cold-drawn and aged	1200	2452.7	6.5

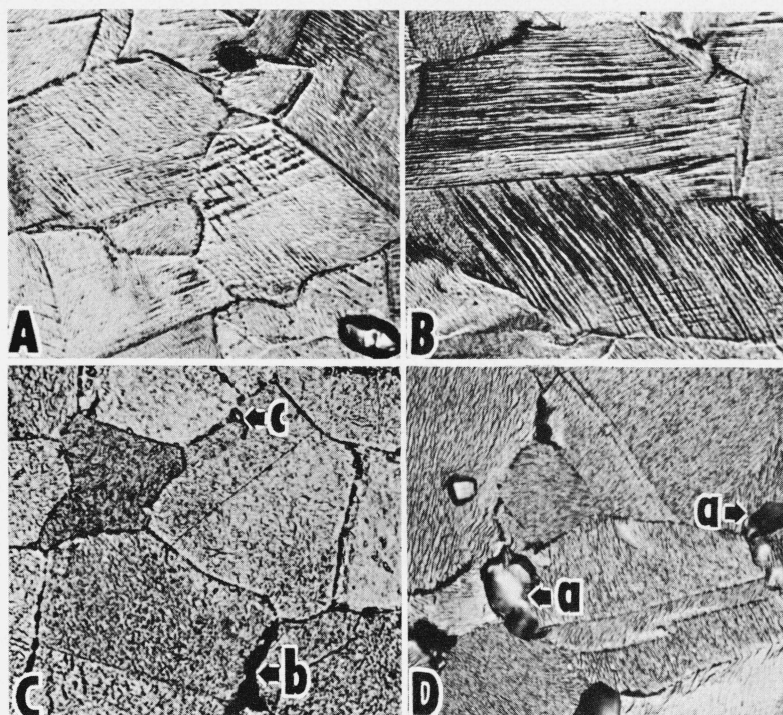


FIGURE 3. *Relation of test conditions to structures of Duranickel after fracture.*
Longitudinal sections, at axis, near fracture. Etched in a solution containing 25 ml each of HNO_3 , glacial acetic acid, HCl and water and 3 g of NaOH ; X 667. Arrows indicate intercrystalline cracks at "a", "b", and "c".

Condition	Test temperature	Fracture time	Reduction of area
A Annealed and aged	°F 700	hr 1367.2	% 5
B Cold-drawn and aged	700	1068	13.5
C Annealed and aged	1200	2059.7	6
D Cold-drawn and aged	1200	2452.7	6.5

were observed in the specimens that had been cold-worked prior to aging. If it is assumed that a greater number of precipitates were initially present in the cold-drawn and aged metal than in the annealed and aged specimens, these additional precipitates could act as sources for the formation of the cracks as illustrated by the models shown in a previous paper [1, figs. 9 and 10]. The effect of cold-drawing prior to aging on the nature of the substructures within the grains of the fractured specimens is shown in figure 3. Intersecting strain markings were evident in the specimens tested at 700 °F (fig. 3A and B); however, a greater number of curved, wavy markings was observed in the cold-drawn and aged metal (fig. 3B), supporting the hypothesis that more precipitates occurred in the cold-

drawn metal. Some of the markings did not traverse the grain from one boundary to the other. Precipitates, subgrain boundaries, and slip bands acted as barriers to the continuous motion of dislocations across the grains as will be illustrated later (fig. 5A) by electron microscopy. A fine structure was observed within the grains of specimens tested at 1200 °F (fig. 3C and D). As will be shown later (figs. 6 and 7) some of this substructure consists of overaged precipitates. Intercrystalline cracks are larger for the cold-drawn and aged specimen (fig. 3Da) than for the annealed and aged specimen (fig. 3Cb). Smaller cracks within the vicinity of the precipitates at the grain boundaries (fig. 3Cc) indicate that voids are nucleated first in these regions, then coalesce to form holes and cracks.

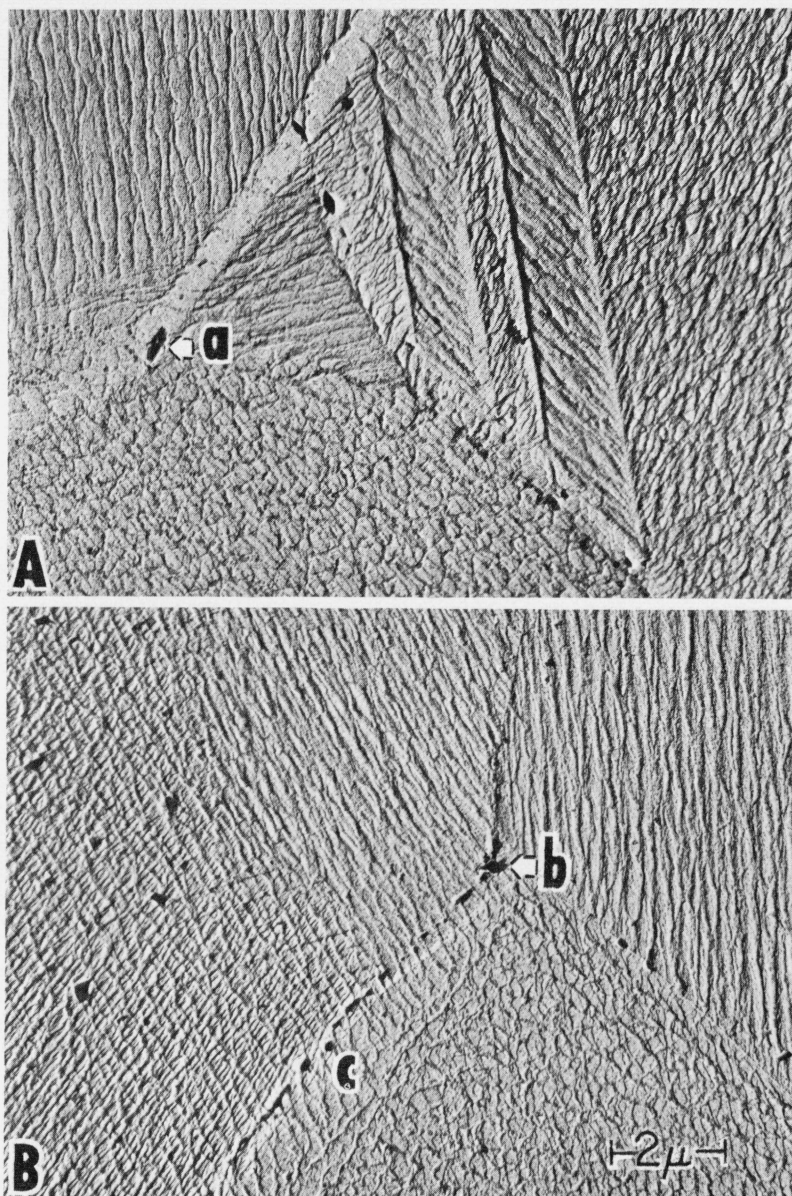


FIGURE 4. *Electron micrographs showing structure of an annealed and aged alloy specimen after fracture in 1367.2 hr at 700 °F.*

Longitudinal sections, near fracture; etched in a solution containing 25 ml each of HNO_3 , glacial acetic acid, HCl and water and 3 g of NaOH ; X 7,500. Arrows indicate Ni_3Al precipitates at the triple points "a" and "b". "c" indicates substructure near grain boundary.

Electron microscopy replica techniques were used to ascertain more fully the size, number and distribution of the precipitates, subgrains, and strain markings in these specimens. The results are shown in figures 4 to 7. Microstructures of the annealed and aged specimen tested at 700 °F (fig. 4) indicated the presence of annealing twins (fig. 4A), well-defined subgrains, precipitates within some of the grains and an absence of them in other grains. It is probable that most were

too small to be resolved at this magnification. Precipitates are also evident at the grain boundaries and appear to be largest at the triple points (fig. 4Aa and Bb). Due to differences in structure of adjacent parent grains, the shape of the subgrains near the boundary is different from that in the interior of the grains (fig. 4Bc). When the microstructures shown in figure 4 are compared with those of the cold-drawn and aged metal, shown in figure 5, it appears that testing

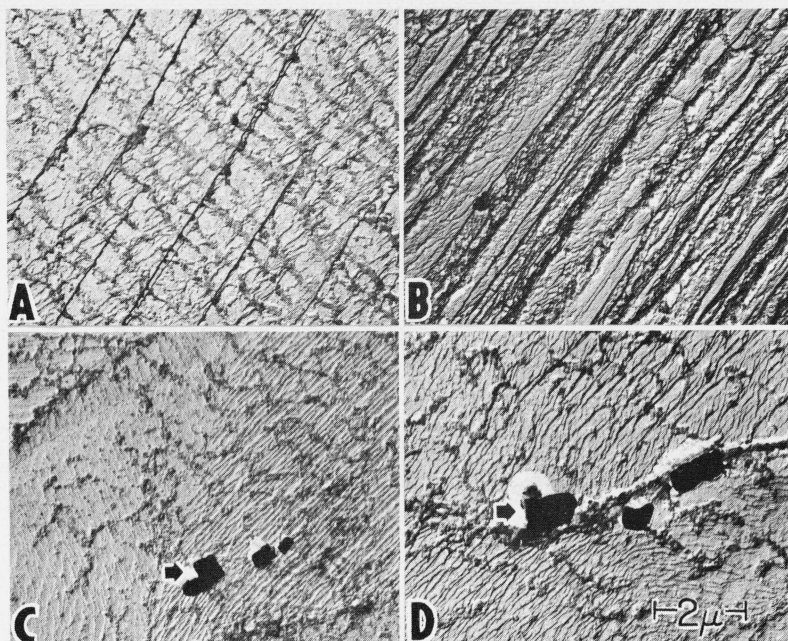


FIGURE 5. *Electron micrographs showing structure of a cold-drawn and aged alloy specimen after fracture in 1068 hr at 700 °F.*

Longitudinal sections, near fracture; etched in a solution containing 25 ml each of HNO₃, glacial acetic acid, HCl and water and 3 g of NaOH; X 6,000. Arrows indicate Ni₃Al precipitates.

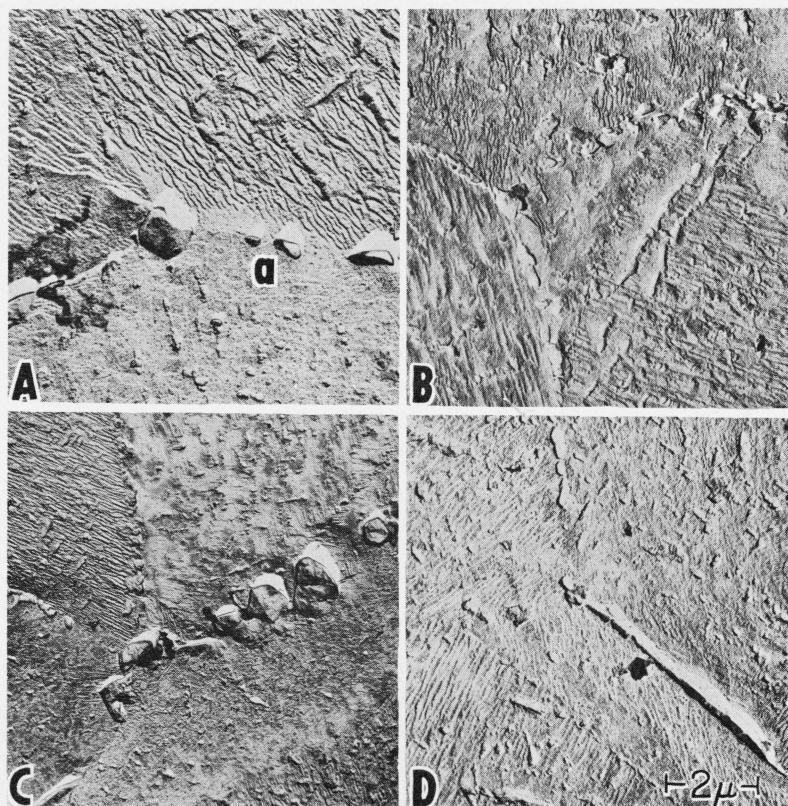


FIGURE 6. *Electron micrographs showing structure of an annealed and aged alloy specimen after fracture in 2059.7 hr at 1200 °F.*

Longitudinal sections, near fracture; etched in a solution containing 25 ml each of HNO₃, glacial acetic acid, HCl and water and 3 g of NaOH; X 6,000. "a" indicates precipitate-free region near grain boundary.

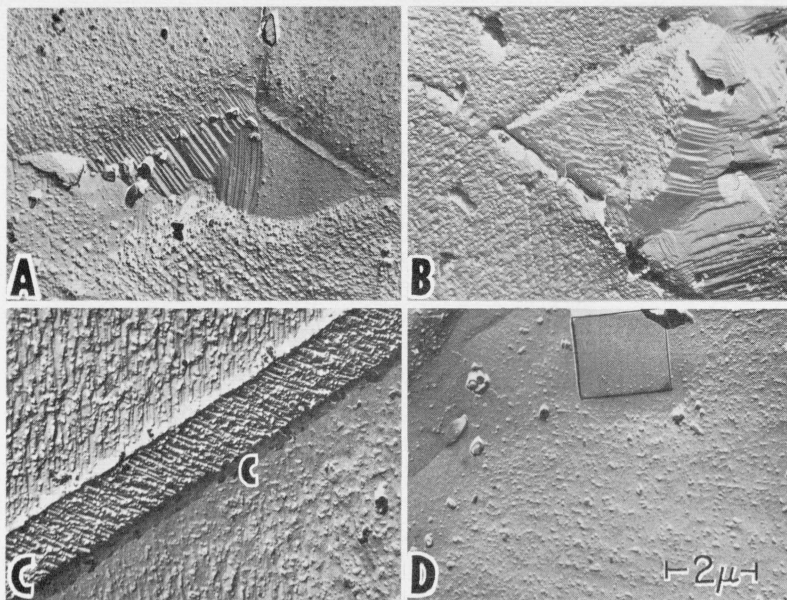


FIGURE 7. Electron micrographs showing structure of a cold-drawn and aged Duranickel specimen after fracture in 2452.7 hr at 1200 °F. Logitudinal sections, near fracture; etched in a solution containing 25 ml each of HNO₃, glacial acetic acid, HCl and water and 3 g of NaOH; X 6,000. "c" indicates precipitate-free region near twin boundary.

the aged metal at 700 °F increases the width of the subgrain boundaries (fig. 5A and C), the size of some of the precipitates (fig. 5C and D, arrows) and the number of slip bands (fig. 5B). The discontinuous nature of some of the strain markings is illustrated in figure 5A. The differences in microstructures had a marked effect on the creep behavior at 700 °F as indicated by the data in figures 8 to 11.

In general, it was found that the size of the precipitates increased with increase in temperature and with increase in time. The size of the precipitates, observed in specimens tested at 1200 °F, was less for the cold-drawn and aged (fig. 7) than for the annealed and aged specimens (fig. 6). Some areas adjacent to the grain boundaries (fig. 6Aa) or twin boundaries (fig. 7Cc) are comparatively free of precipitates. However, precipitation within the grains appears to have taken place on definite preferred planes and the size of these precipitates was less affected by cold-drawing than the size of those at the grain boundary. These differences in microstructures had little effect on the creep behavior at 1200 °F as indicated by figures 9 and 10.

4. Test Data and Discussion

Davies, Richards, and Wilshire [3] studied the effect of prior cold-work on creep and fracture of a nickel-0.1 atomic percent gold alloy. They concluded that, below the recrystallization temperature, creep rate was reduced and rupture life was increased when the prestrain exceeded the initial strain. Their results

were interpreted on the basis of varying contributions from the grain interiors and the grain boundary regions. Dennison and Wilshire [4] carried out tests on three grades of nickel and observed that decrease in purity was accompanied by an increase in creep resistance and rupture life. They concluded that impurities impeded grain boundary migration, caused a decrease in ductility and an increase in the occurrence of cracking. The effects of cold-drawing on creep of some nickel-copper alloys were discussed by Jenkins and Willard [5].

Strain-time curves for some of the creep specimens used in the present investigation are shown in figure 8. Cold-drawing has reduced the extent (time and strain) of both the first and second stages of creep below the values for the annealed specimens, previously reported [2]. For the aged material the first stage was practically nonexistent. The shape of the curves are both temperature and stress dependent. Time lag before the initiation of plastic deformation at 1200 °F is believed to be due in part to the blocking of the motion of dislocations by the precipitates that formed at 1200 °F before loading the specimens. These precipitates have been shown both by x-ray and electron diffraction to be predominantly particles of Ni₃Al. The properties of this compound have been studied extensively by Guard and Westbrook [6].

The relation between stress and second stage creep rate³ is shown in figure 9. A linear relationship be-

³The second stage creep rates were obtained from strain-time curves when the data were plotted on a linear scale (not shown).

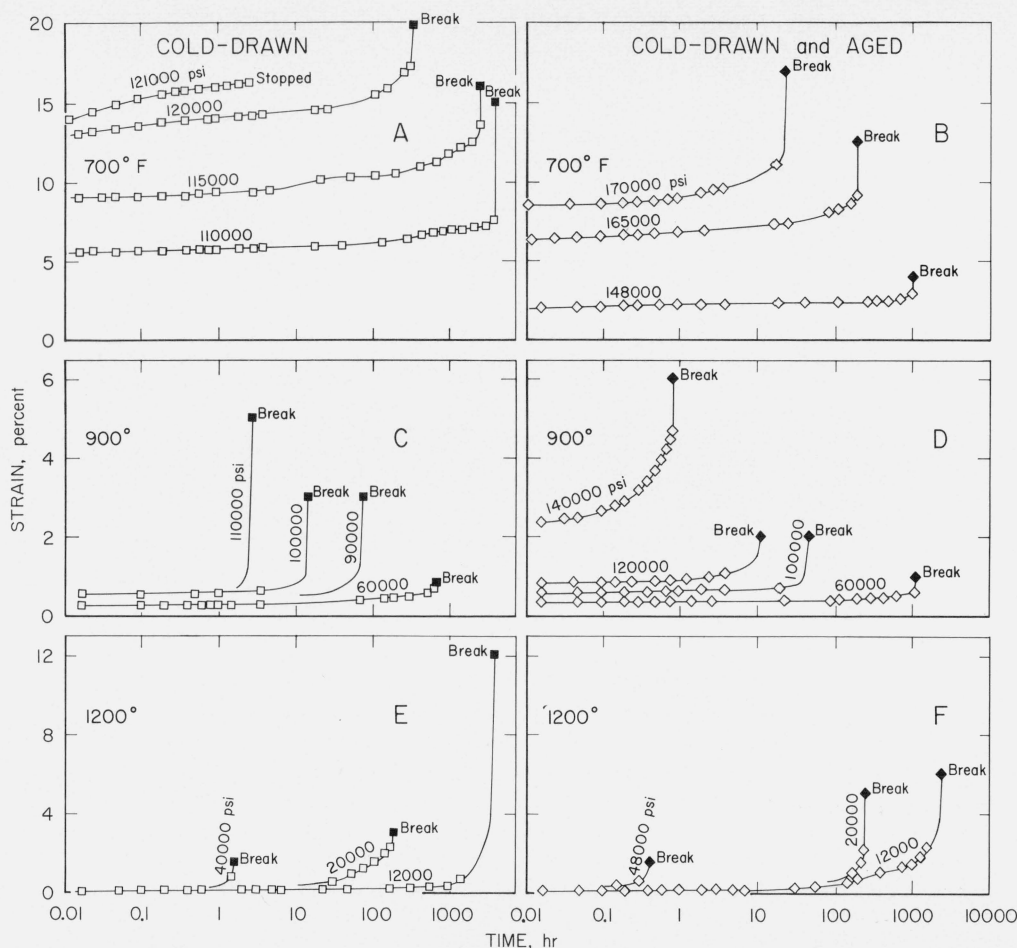


FIGURE 8. Strain-time relations of cold-drawn and cold-drawn and aged alloy specimens tested at different temperatures.

tween these variables was not obtained when the data were plotted on linear, log-log, or semilog coordinates. The sigmoidal form of some of the curves indicates the existence of a creep rate for which strain hardening is a maximum. Cold-drawing the metal increased the strength at 700 and 900 °F with the greatest increase occurring at the faster rates (fig. 9A). Furthermore, cold-drawing prior to aging improved the creep strength (fig. 9B) in comparison to the annealed and aged metal. No significant difference in strength is observed at 1200 °F where recrystallization and overaging occurred during the tests.

The effect of stress on time to fracture is shown in figure 10. Obviously, no simple equation could be used to describe the relationship between these variables or between the family of curves for specimens having the same or different heat-treatments. It is observed, at 700 and 900 °F, that strengthening due to cold-drawing or aging is less effective at the longer test times than at the shorter times. Recovery of the strained material is believed to be the major cause of this phenomenon. Although the stress-creep rate

and stress-fracture time curves appear to be similar, no consistent relation existed between creep rates and fracture times for either the cold-drawn or cold-drawn and aged specimens.

The interrelationship between second stage creep rate and ductility is shown in figure 11. In general, elongation and reduction of area values increased with increase in creep rate at 700 °F and decreased with increase in creep rate at 1200 °F. A large number of mechanisms, such as strain hardening, recovery, aging, transcrystalline and intercrystalline cracking, accompanied creep at 900 °F and caused the ductility values to vary in the same manner as was observed for annealed and annealed and aged Duranickel [2].

Rockwell hardness tests were made at room temperature on specimens after fracture to ascertain the effect of creep on hardening. Hardness indentations were made on cross sections of both the shoulder and on the deformed (reduced) section near fracture of the cold-drawn and cold-drawn and aged specimens in order to ascertain the effect of plastic deformation on aging characteristics. Small load-free control

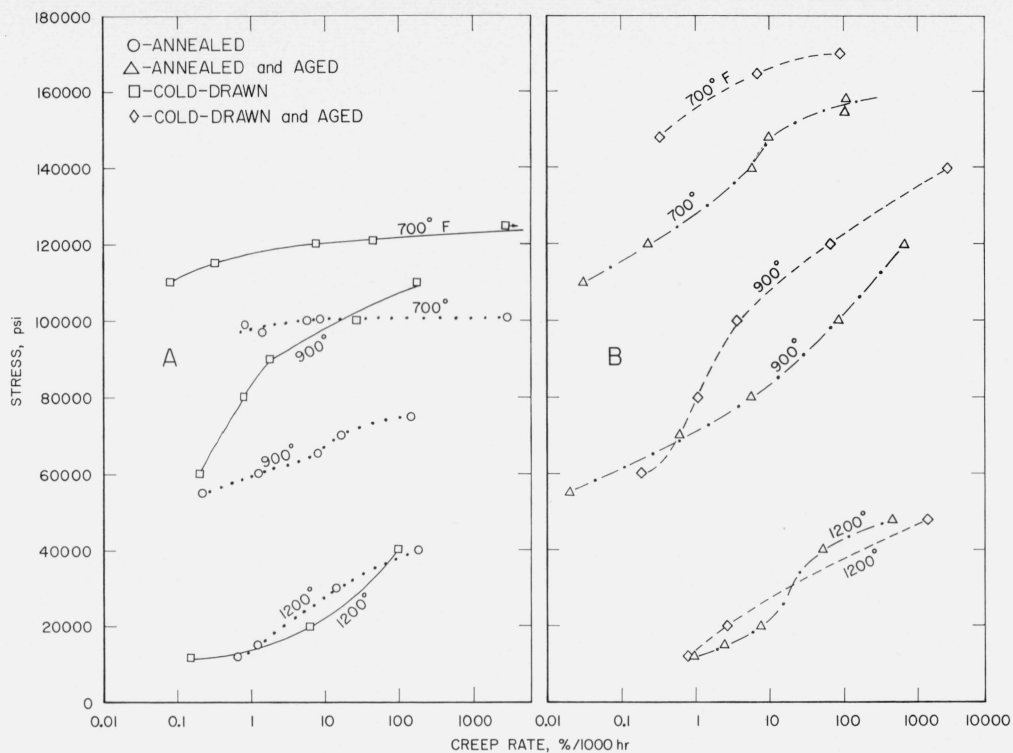


FIGURE 9. Effect of cold-drawing and aging on the stress-second stage creep rate relations of the alloy.

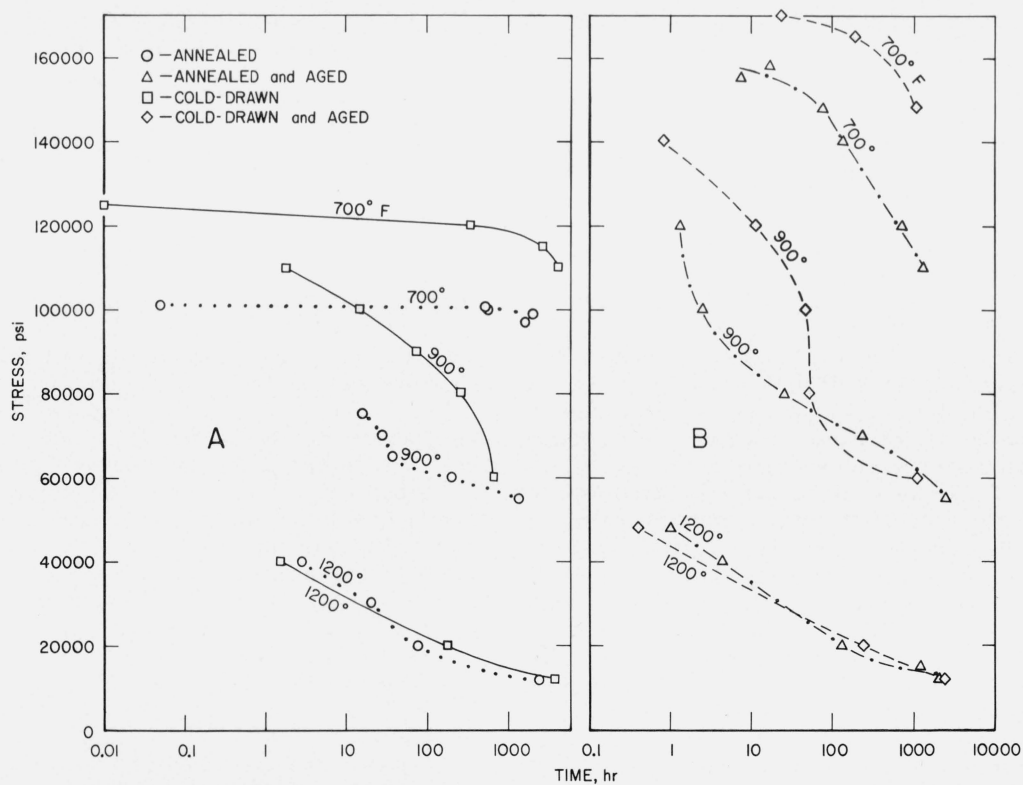


FIGURE 10. Effect of cold-drawing and aging on the stress-fracture time relations of the alloy.

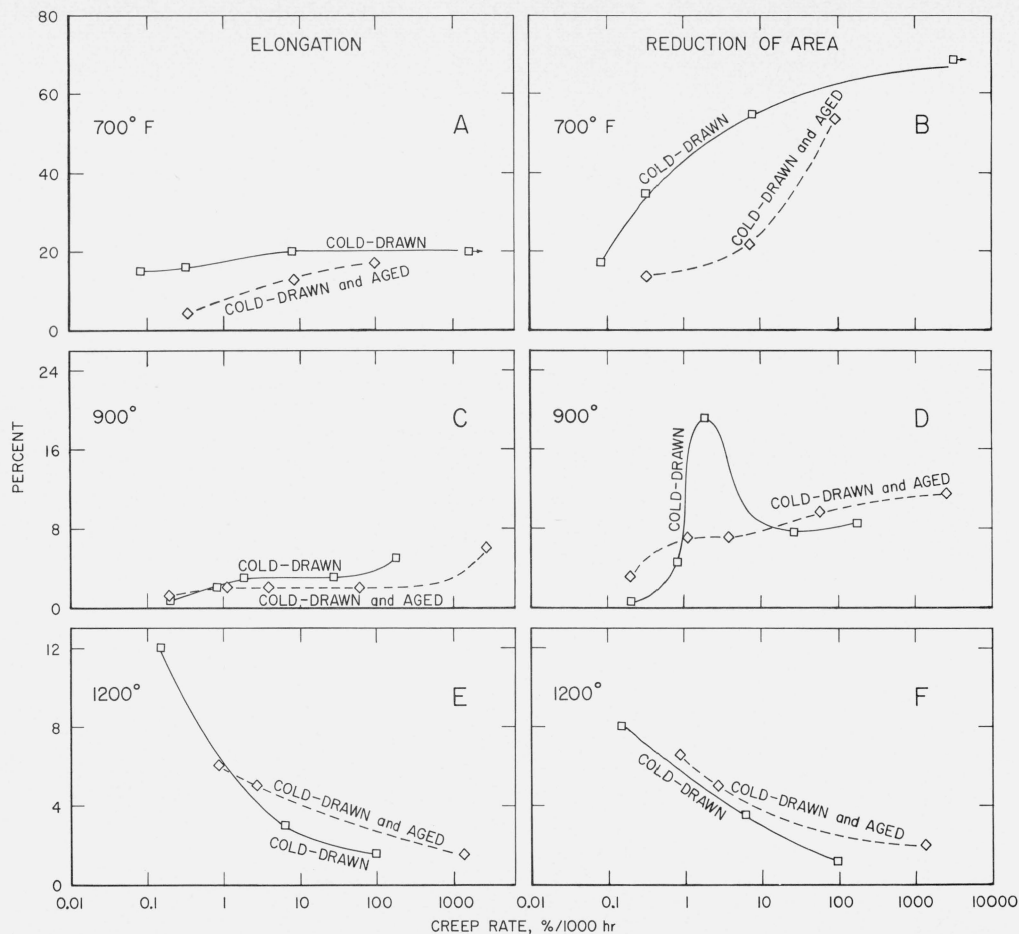


FIGURE 11. Influence of aging on the ductility-second stage creep rate relations of cold-drawn alloy specimens tested to fracture at different temperatures.

samples, heat-treated for times corresponding to the various fracture times in the creep tests, were tested for hardness and used for comparison purposes. As indicated by the hardness data for the cold-drawn specimens (fig. 12A, C, and E) precipitation-hardening was more predominant at 900 than at 700 °F. This was evident for the specimens exposed to temperature only (fig. 12A), for unstrained portions of specimens (shoulders) exposed to temperature and stress (fig. 12C), and for the strained portions of the specimens (reduced sections) tested to fracture in creep (fig. 12E). Extensive recovery, recrystallization, and overaging occurred in the specimens tested at 1200 °F as shown by the decrease in hardness with increase in test time. This behavior was also evident for the cold-drawn and aged specimens (fig. 12B, D, and F) tested at 1200 °F; however, test time had only a slight effect on hardness values of the specimens tested to fracture at 700 and 900 °F. These data tend to confirm the general trends associated with the hardness values obtained for the load-free specimens and shown in figure 1.

5. Summary

Creep tests were made at temperatures of 700, 900, and 1200 °F (644, 755, and 921 °K) on a nickel-4.2 percent aluminum alloy initially as cold-drawn and as cold-drawn and age-hardened.

Microstructures and test data obtained in the present investigation are compared to results shown previously for the annealed, and annealed and aged alloy.

The hardness of the alloy was increased by cold-drawing or aging. Additional hardening was induced by aging subsequent to the cold-drawing operation.

Due to the existence and formation of numerous barriers to the motion of dislocations through the crystal lattice, cold-drawing and aging increased the creep strength and decreased the ductility of the alloy at 700 and 900 °F. No significant effect was apparent at 1200 °F, which is slightly above the recrystallization temperature.

Elongation and reduction of area values increased with increase in creep rate at 700 °F and with decrease in creep rate at 1200 °F.

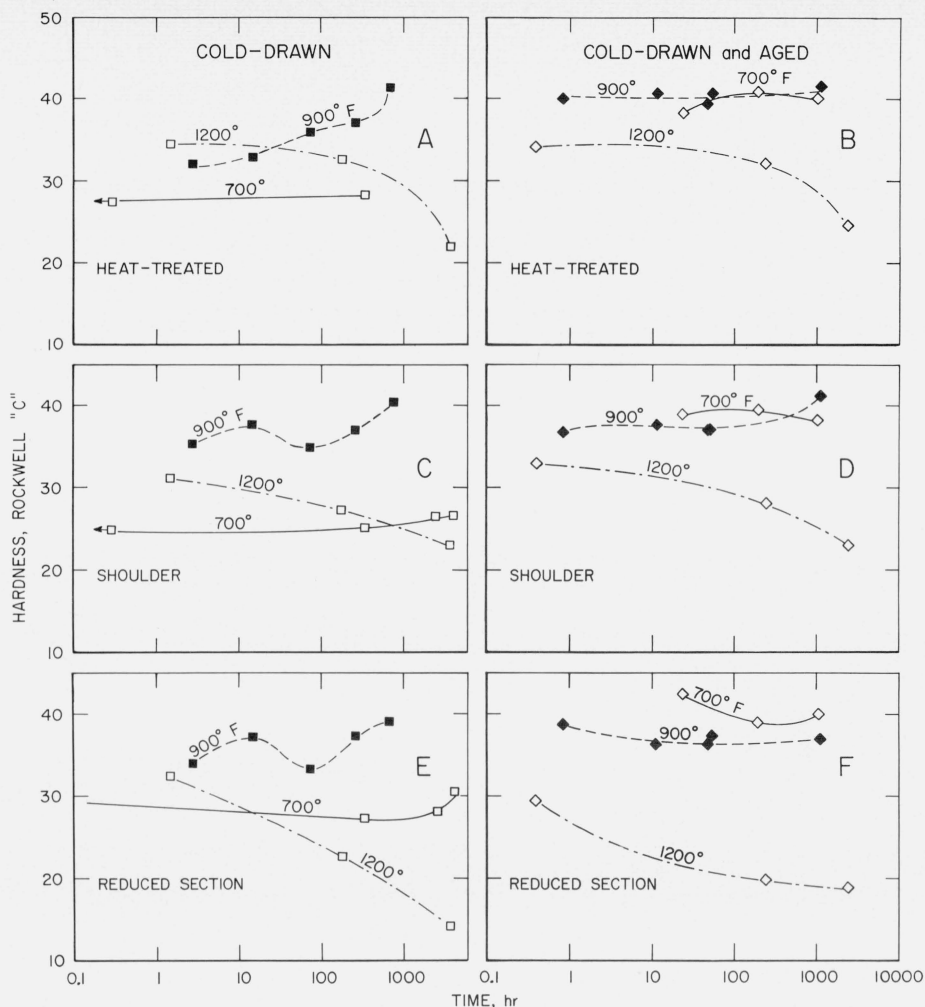


FIGURE 12. Effect of test temperature and time on hardness, at room temperature, of cold-drawn and cold-drawn and aged alloy specimens.

A, B, load-free control specimens as heat-treated; C, D, E, F, creep specimens after fracture.

Aging the cold-drawn alloy made it more ductile at 1200 °F and less at 700 °F.

Post-test hardness values decreased with increase in test time at 1200 °F, due to recrystallization. With few exceptions, the hardness after testing at 900 °F was equal to or greater than that after testing at 700 °F.

The tendency toward intercrystalline cracking increased with increase in test temperature. The number of cracks was increased by cold-drawing prior to age-hardening.

Microstructures, associated with high strength and ductility at the lower temperatures indicated the existence of small precipitated particles, small subgrains and rather straight intersecting and nonintersecting strain markings. However, at 1200 °F, few markings were evident and coalesced precipitates appeared predominantly in the grain boundaries. The shape and number of the precipitates appeared to depend on where they are located in the crystal lattice.

(Paper 70C3-231)

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6. References

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